
तार और बिटुमिनी सामग्री की
परीक्षण विधियाँ — टोल्यूनि में अघुलनशील
सामग्री का ज्ञात करना
(दूसरा पुनरीक्षण)

**Methods for Testing Tar and
Bituminous Materials —
Determination of Matter Insoluble
in Toluene**
(Second Revision)

ICS 75.140

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Bitumen, Tar and Related Products Sectional Committee, had been approved by the Petroleum, Coal and Related Product Division Council.

This standard was originally published in 1958 as 'Methods for testing tar and bituminous materials — Determination of matter insoluble in toluene' and first revised in 1978. 'Methods for testing tar and bituminous materials' was originally published as series of 22 standards in the form of a booklet, as listed below:

| <i>IS No.</i> | <i>Title</i> |
|------------------------|---|
| 1201 : 2004 | Sampling |
| 1202 : 1978 | Determination of specific gravity |
| 1203 : 1978 | Determination of penetration |
| 1204 : 1978 | Determination of residue of specified penetration |
| 1205 : 1978 | Determination of softening point |
| 1206 (Part 1) : 1978 | Determination of viscosity: Part 1 Industrial viscosity |
| 1206 (Part 2) : 1978 | Determination of viscosity: Part 2 Absolute viscosity |
| 1206 (Part 3) : 1978 | Determination of viscosity: Part 3 Kinematic viscosity |
| 1207 : 1978 | Determination of equiviscous temperature (EVT) |
| 1208 : 1978 | Determination of ductility |
| 1209 : 1978 | Determination of flash point and fire point |
| 1210 : 1978 | Float test |
| 1211 : 1978 | Determination of water content dean and stark method |
| 1212 : 1978 | Determination of loss on heating |
| 1213 : 1978 | Distillation test |
| 1214 : 1978 | Determination of matter insoluble in benzene (<i>WITHDRAWN</i> due to toxic nature of benzene) |
| 1215 : 1978 | Determination of matter insoluble in toluene |
| 1216 : 1978 | Determination of solubility in carbon disulphide trichloroethylene |
| 1217 : 1978 | Determination of mineral matter ash |
| 1218 : 1978 | Determination of phenols |
| 1219 : 1978 | Determination of naphthalene |
| 1220 : 1978 | Determination of volatile matter content |

However, the Committee responsible for the formulation of standards in the field of bitumen, tar and related products decided to publish these Indian standards separately for each test so as to make it user friendly. Accordingly, second revision of the standard, IS 1215 'Methods for testing tar and bituminous materials — Determination of matter insoluble in toluene' was taken up to formulate individual standard.

In this revision, the following modification have been made:

- a) References have been updated

The Composition of the Committee responsible for formulation of this standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this Standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR TESTING TAR AND BITUMINOUS MATERIALS — DETERMINATION OF MATTER INSOLUBLE IN TOLUENE

(*Second Revision*)

1 SCOPE

This standard prescribes methods of test for determination of matter insoluble in toluene for road tar and pitch.

2 REFERENCES

The following standard contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreement based on the standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

| <i>IS No.</i> | <i>Title</i> |
|---------------|--|
| 537 : 2011 | Toluene — Specification (<i>second revision</i>) |

3 METHOD FOR SOFT GRADE PITCH

3.1 Apparatus

3.1.1 Beaker — 200 ml capacity.

3.1.2 Sintered Glass Crucible — Porosity No. 4.

3.1.3 Filter Papers — Two, each about 150 mm in diameter, grade Whatman No. 5 or its equivalent.

3.1.3.1 Filtering funnel and bottle

3.1.4 Water Bath — Maintained to constant temperature of 95 ± 5 °C. Glycerin shall be use in place of water for temperature 95 °C and above.

3.1.5 Graduated Cylinder — 100 ml capacity.

3.1.6 Analytical Balance

3.2 Reagent

3.2.1 Toluene, conforming to IS 537.

3.3 Procedure

3.3.1 Take accurately weighed 2 g of the thoroughly mixed material in a 200 ml beaker and warm it on a water bath. Add 100 ml of hot toluene at 95 ± 5 °C and mix intimately. After settling for 20 min, with the beaker standing on the water bath, decant the supernatant liquid

cautiously. Avoiding disturbance of the sediment, either through filter papers or sintered glass crucible.

3.3.1.1 Method using filter papers

Counterpoised double filter papers shall be used. For counterpoising, heat two filter papers to a temperature of 100 ± 2 °C and reduce them to equal weight by removing the apex of the heavier paper after folding. For filtering, use the uncut paper inside the originally heavier paper, so that separated material is retained by the inner paper, yet both of them are equally subjected to any action exerted by toluene. Wash the residue by repeated decantation with toluene heated to 95 ± 5 °C until 250 ml of toluene has been used in all. Transfer the residue to the filter paper and continue the washing until altogether 400 ml of hot toluene has been used. Dry the residue in an oven at a temperature of 100 ± 2 °C until it is of constant weight.

3.3.1.2 Method using sintered glass crucible

Dry the crucible at a temperature of 100 ± 2 °C and weigh accurately when cooled. Decant the toluene solution through the crucible using gentle suction to assist filtration and avoiding the transfer of the sediment. Wash repeatedly by decantation until 250 ml of toluene has been used. Transfer the sediment in the beaker to the crucible and continue washing until 400 ml of toluene has in all been used. Dry the residue in an oven at a temperature of 100 ± 2 °C until it is of constant weight.

3.3.2 Preserve the residue for ash determination [see IS 1217 'Methods for testing tar and bituminous materials: Determination of mineral matter (Ash)'].

3.4 Calculation

Calculate and express the result as percentage by mass of the material taken for the test as follows:

Insoluble matter in toluene, percent by mass =

$$\frac{W_1}{W_2} \times 100$$

where

W_1 = mass in g, of the dry material (test sample);
and

W_2 = mass in g, of insoluble matter (or residue).

4 METHOD FOR SOFT MEDIUM, HARD MEDIUM AND HARD PITCH GRADES

4.1 Procedure

4.1.1 Treat about 1 g of the material, finely divided, if possible, and accurately weighed, with 100 ml of toluene at the laboratory temperature, in the manner described under **3.3.1**.

4.1.2 Treat the residue in the beaker in exactly the same manner as described in **3.3.1** with a further addition of 100 ml of toluene at laboratory temperature. Then treat it with three successive quantities of toluene at 95 ± 5 °C, each of 100 ml, in exactly the same manner as described in **3.3.1** except that each settling period during which the beaker is standing in the water bath need only be of 5 min.

4.1.3 Transfer the residue to the filter paper or sintered glass crucible and continue the washing until a further 400 ml of hot toluene has been used. Dry the residue in an oven at a temperature of 100 ± 2 °C until it is of constant mass.

4.1.4 Preserve the residue for ash determination [see IS 1217] 'Methods for testing tar and bituminous materials: Determination of mineral matter (Ash) (first revision)'].

4.2 Calculation

Calculate and express the result as percentage by mass of the material taken for the test as follows:

Insoluble matter in toluene, percent by mass =

$$\frac{W_1}{W_2} \times 100$$

where

W_1 = mass in g, of the dry material (test sample);
and

W_2 = mass in g, of insoluble matter (or residue).

5 REPORT

Report the percentage insoluble matter in toluene to the nearest 0.1 percent.

6 PRECISION

6.1 The duplicate test results shall not differ by more than the following:

| Repeatability | Reproducibility |
|---------------|-----------------|
| 0.5 percent | 1.0 percent |

7 PRECAUTIONS

7.1 The solvent toluene is extremely inflammable. Handling of toluene shall be carried out away from any source of heat or flames.

7.2 Toluene has considerable health hazards. Refer to the Material Safety Data Sheet (MSDS) of toluene for necessary handling precautions.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Bitumen, Tar and Related Products Sectional Committee, PCD 06

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